

REPORT

DETERMINATION OF THE SURFACE TENSION OF AN AQUEOUS SOLUTION OF



NOTOX Project 338592
NOTOX Substance 111834/C

CONFIDENTIALITY STATEMENT

This report contains the unpublished results of research sponsored by [REDACTED]
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STATEMENT OF GLP COMPLIANCE

NOTOX B.V., 's-Hertogenbosch, The Netherlands

The study described in this report has been correctly reported and was conducted in compliance with the most recent edition of:

The OECD Principles of Good Laboratory Practice

which are essentially in conformity with:

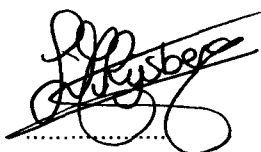
The United States Food and Drug Administration. Title 21 Code of Federal Regulations Part 58.

The United States Environmental Protection Agency (FIFRA). Title 40 Code of Federal Regulations Part 160.

The United States Environmental Protection Agency (TSCA). Title 40 Code of Federal Regulations Part 792.

Study Director

L.M. van Rijsbergen

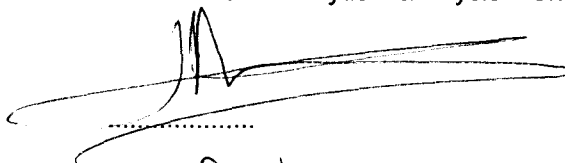


Date: April 08, 2002.

Management

Dr. Ir. H. Willems

Section Head Analytical & Physical Chemistry



Date: April 08, 2002

QUALITY ASSURANCE STATEMENT

NOTOX B.V., 's-Hertogenbosch, The Netherlands

This report was audited by the NOTOX Quality Assurance Unit to ensure that the methods and results accurately reflect the raw data.

The dates of Quality Assurance inspections and audits are given below.
During the on-site inspections procedures applicable to this type of study were inspected.

DATES OF QAU INSPECTIONS/AUDITS

REPORTING DATES

on-site inspection (s)

03-Dec-2001 to 05-Dec-2001 (process)

11-Dec-2001

protocol inspection (s)

14-Jan-2002 (study)

14-Jan-2002

report audit (s)

21-Mar-2002 (study)

21-Mar-2002

Head of Quality Assurance
C.J. Mitchell B.Sc.

RR

.....A. Morgan

Date: 10 April, 2002

SUMMARY

The determination of the surface tension of an aqueous solution of [REDACTED] was based on EEC-Directive 92/69 EEC, A.5 "Surface tension" (1992) and OECD guideline no. 115: "Surface tension of aqueous solutions", July 27, 1995.

A (saturated) solution of [REDACTED] was prepared by stirring 98.9 mg test substance with 100 ml water for approximately 21 hours. During the stirring period, the test vessel was securely closed to prevent the volatile test substance from evaporation. After stirring, no (liquid) test substance was observed. Therefore, the clear solution was used for testing without dilution with water.

The surface tension of the prepared solution of [REDACTED] in water (0.989 g/l) is 52.6 mN/m. Based on the criteria as outlined in the guideline, it is concluded that [REDACTED] should be regarded as a surface active material.

Temperature of measurement was $20.0 \pm 0.5^{\circ}\text{C}$.

PREFACE

Sponsor	[REDACTED] [REDACTED] [REDACTED] [REDACTED]
Study Monitor	Dr. C.L.J. Braun SHERA, Regulatory Affairs
Testing Facility	NOTOX B.V. Hambakenwetering 7 5231 DD 's-Hertogenbosch The Netherlands
Study Director	L.M. van Rijsbergen
Study Plan	Start : 04 February 2002 Completed : 05 February 2002

TEST SUBSTANCE

Identification	[REDACTED]
Chemical name	[REDACTED]
CAS RN	[REDACTED] [REDACTED]
Description	Clear colourless liquid
Batch	1510-14
Purity	See Certificate of Analysis
Test substance storage	In refrigerator in the dark
Stability under storage conditions	Stable
Expiry date	01 January 2003

The sponsor is responsible for all test substance data unless determined by NOTOX.

Note: **Don't heat up the test substance above 50°C**

PURPOSE

The purpose of the study was to evaluate the surface tension of the test substance in an aqueous solution, by means of a ring tensiometer.

GUIDELINES

The study procedure described in this report is based on the following guidelines:

European Economic Community (EEC), EEC-Directive 92/69 EEC, Part A, Methods for the determination of physico-chemical properties, A.5 "Surface tension", EEC Publication No. L383, December 1992.

Organization for Economic Co-operation and Development (OECD), OECD guidelines for Testing of Chemicals, guideline no. 115: "Surface tension of aqueous solutions", July 27, 1995.

ARCHIVING

NOTOX B.V. will archive the following data for at least 10 years: protocol, report, test substance reference sample and raw data. Thereafter, no data will be withdrawn without the sponsor's written consent.

TEST SYSTEM AND RATIONALE

Test system	The experiment was carried out using a tensiometer (model K6; Krüss GmbH, Hamburg, FRG). The measuring body is a platinum-iridium ring with a wire radius of 0.185 mm and a ring radius of 9.545 mm.
Water	LiChrosolv; Merck, FRG
Conditions	Measurements were performed at a temperature of $20.0 \pm 0.5^\circ\text{C}$.
Rationale	Recognized by the international guidelines as recommended test system (EEC, OECD).

VALIDATION OF THE TEST PROCEDURE

The test procedure as outlined in this report is validated periodically, using n-hexane (LiChrosolv; Merck, FRG). The results are in accordance with the criteria of OECD Guideline No. 115 "Surface tension of aqueous solutions", July 27, 1995.

PERFORMANCE OF THE TEST

Preparations before the measurement

All glassware used was washed with chromo-sulphuric acid. The measurement vessel was washed with hot chromo-sulphuric acid and subsequently with syrupy phosphoric acid. Thereafter all glassware was thoroughly rinsed in tap-water and finally washed with water. The measuring body (ring) was rinsed thoroughly in water, briefly immersed in chromo-sulphuric acid, washed in water and finally briefly heated above a flame.

Calibration of the tensiometer

The apparatus was calibrated using water with established surface tension. The calibration factor Φ_b , by which all the instrument readings are multiplied, was determined in accordance with the equation:

$$\Phi_b = \sigma_o / \sigma_g$$

σ_o = the surface tension of water at the test temperature cited in the literature
(Gebrauchsanweisung für Tensiometer 8451. Krüss GmbH, Hamburg, Germany).

σ_g = measurement value of the surface tension of the water at the test temperature.

Preparation of the test solution

A (saturated) solution of [REDACTED] was prepared by stirring 98.9 mg test substance with 100 ml water for approximately 21 hours (20°C). During the stirring period, the test vessel was securely closed to prevent the volatile test substance from evaporation. After stirring, no (liquid) test substance was observed. Therefore, the clear solution was used for testing without dilution with water.

Test procedure

The cleaned and rinsed measurement vessel was filled with the test solution. The measurement vessel was placed in a thermostated waterbath (20°C) on the mobile sample table. The table was raised until the ring was immersed below the surface of the solution. Subsequently, the table was lowered until the ring was attached to the liquid surface. After some additional lowering of the table, the ring exerts a torque on the force measuring system. This torque was compensated for by turning the large knob on the force measuring system clockwise. Lowering of the table and subsequent compensation of the resulting torque were continued in small increments until the lamina broke, i.e., the upward force on the ring completely overcame the surface tension force and the ring was torn away from the surface. The surface tension was then recorded. After completing the measurement the ring was immersed below the surface again and the measurements were repeated until a constant surface tension value was reached. The time passed since the solution was transferred to the measurement vessel was recorded for each determination.

DATA HANDLING

The surface tension is given as: mN/m (SI sub-unit).

Corrections of the measured values:

In order to calculate the surface tension, the value read on the apparatus was multiplied by the calibration factor Φ_b . This yielded a value which was only approximate, and required additional correction. The additional correction was made using a table compiled on the basis of Harkins-Jordan correction (W.D. Harkins, H.F. Jordan, J. Amer. Chem. Soc. 52, 1751, 1930); see Table 1.

Table 1

Experimental value [mN/m]	Corrected value (water calibration) [mN/m]	Experimental value [mN/m]	Corrected value (water calibration) [mN/m]
20	18.1	50	48.6
22	20.1	52	50.7
24	22.1	54	52.8
26	24.1	56	54.9
28	26.1	58	57.0
30	28.1	60	59.1
32	30.1	62	61.3
34	32.1	64	63.4
36	34.1	66	65.5
38	36.1	68	67.7
40	38.2	70	69.9
42	40.3	72	72.0
44	42.3	74	-
46	44.4	76	-
48	46.5	78	-

According to the criteria as outlined in the EEC Directive, substances showing a surface tension lower than 60 mN/m under the conditions of this method, should be regarded as being surface active materials.

RESULTS

Determination of the calibration factor Φ_0 is described in the Appendix.

Table 2 shows the individual measurements and the corrected surface tension of a solution of test substance in water at a concentration of 0.989 g/l.

Table 2 Surface tension of an aqueous solution of [REDACTED]

Time passed between preparation and the first measurement:
21 hours stirring time and 24 minutes equilibration time.

Determination	Time expired ¹ [min]	Surface tension ² [mN/m]			
		measured	mean not corrected	mean corrected for calibration	mean corrected using Harkins & Jordan, 1930
1	24	50.5	52.3	53.8	52.6
2	25	52.2			
3	26	52.1			
4	27	52.5			
5	28	52.2			
6	29	52.3			

¹ Time passed since the solution was transferred to the measurement vessel.

² Because the surface tension of a test solution in the measurement vessel alters over a period of time, several measurements were performed until a state of equilibrium was reached. The mean value was calculated from measurement 2 - 6.

In conclusion, the surface tension of the prepared solution of [REDACTED] in water (0.989 g/l) is 52.6 mN/m. Based on the criteria as outlined in the guideline, it is concluded that [REDACTED] should be regarded as a surface active material.

APPENDIX

Determination of the calibration factor Φ_b

Surface tension of water σ_g (mN/m): (measured at 19.8°C)	1. 66.1 2. 68.6 3. 70.5 4. 70.6 5. 70.8 6. 70.6 7. 70.5
Mean surface tension of water (mN/m):	70.6

Several measurements were performed until a state of equilibrium was reached. The mean value was calculated from measurement 3-7.



$\sigma_o = 72.62$ mN/m (Krüss GmbH, at 19.8°C)

$\Phi_b = \sigma_o / \sigma_g = 72.62 / 70.6 = 1.029$.



CERTIFICATE OF ANALYSIS

Certificate of AnalysisTNA-2001007
page 1 of 2

ICS-331

Product name : 
Chemical name : 
Batch number : 1510-14

Test results:

Method	Analysis of	Unit	Result *1
Jo/72.11, Jo/95.2	Peroxidic compounds (sum) <i>See page 2 for a specification</i>	% m/m	28.6 (± 1.5)
J20010792		% m/m	67.0 (± 1.0)
J20010792		% m/m	2.0 (± 0.3)
Amp/88.9	Water	% m/m	2.6 (± 0.3)
J20010792	Unidentified impurities	% m/m	0.5 (± 0.2)

*1 bracketed values are estimated 95% confidence intervals

File code : TNA-2001007

Analytical documentation : 20010792

[Redacted]

[Redacted]

Certificate of Analysis

[Redacted]

TNA-2001007
page 2 of 2

[Redacted]atch 1510-14: specification of the peroxidic compounds

structure	% m/m
[Redacted]	

2

[Redacted]